

Claims

- 1 Crystalline salts of 5-methyl-(6R,S)-, -(6S)- and -(6R)-tetrahydrofolic acid.
- 5 2 Crystalline salts of 5-methyl-(6S)- and -(6R)-tetrahydrofolic acid.
- 3 The crystalline calcium salt of 5-methyl-(6S)- and -(6R)-tetrahydrofolic acid.
- 4 The crystalline calcium salt of 5-methyl-(6S)-tetrahydrofolic acid with 2 theta values of 6.5, 13.3, 16.8 and 20.1.
- 10 Sub 10
A2 5 The crystalline calcium salt of 5-methyl-(6S)-tetrahydrofolic acid with 2 theta values of 5.3, 6.9, 18.7 and 21.1.
- 15 6 The crystalline calcium salt of 5-methyl-(6S)-tetrahydrofolic acid with 2 theta values of 6.8, 10.2, 15.4 and 22.5.
- 7 The crystalline calcium salt of 5-methyl-(6S)-tetrahydrofolic acid with 2 theta values of 6.6, 15.9, 20.2 and 22.5.
- 20 8 A method of producing crystalline salts of 5-methyl-(6R,S)-, -(6S)- and 5-methyl-(6R)-tetrahydrofolic acid, characterised in that salts of 5-methyl-(6R,S)-, -(6S)- or -(6R)-tetrahydrofolic acid are crystallised from a polar medium after thermal treatment.
- 25 9 A method according to claim 8, characterised in that crystallisation is effected after thermal treatment at a temperature above 60°C.
- 30 9 A method according to claim 8, ^{wherein the} characterised in that crystallisation is effected after thermal treatment at a temperature above 85°C.

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~~A 10~~ A method according to claim 8, ~~characterised in that~~ ^{wherein the} crystallisation is effected from a solution.

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~~A 11~~ A method according to claim 8, ~~characterised in that~~ ^{wherein the} crystallisation is effected from a suspension.

~~12~~
~~A 12~~ A method according to claims ~~11 or 12~~ ¹⁰, characterised in that crystallisation is effected from water or from a mixture of water and an organic solvent which is miscible with water.

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14 The use of crystalline salts of 5-methyl-(6S)- or -(6R)-tetrahydrofolic acid as constituent for the production of drugs or as a food additive.

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15 Preparations containing crystalline salts of 5-methyl-(6S)- or -(6R)-tetrahydrofolic acid.

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Table 1: Crystalline calcium salt of 5-methyl-(6S)-tetrahydrofolic acid (Type I)

Diffractometer : Transmission
 Monochromator : Curved Ge(111)
 Wavelength : 1.540598 Cu
 Detector : Linear PSD
 Scan Mode : Debye-Scherrer / Moving PSD / Fixed omega
 2Theta scan

! Peak search parameters : Expected halfwidth : .150
 ! Significance level : 2.5
 ! Peak height level : 10

Peaklist [Range 1 : 2Theta = 5.000 34.980 .020 Imax = 765]

!	D	2Theta	I(rel)	I(abs)	FWHM	h	k	l
13.474630	6.5544	100.0	755	.2200				
8.979750	9.8420	18.5	140	.1600				
6.936035	12.7526	20.3	153	.1600				
6.662427	13.2786	38.3	289	.0800				
6.497896	13.6164	29.4	222	.1200				
6.323596	13.9935	18.8	142	.0200				
6.148863	14.3933	14.0	106	.0400				
5.966675	14.8352	15.5	117	.1200				
5.593548	15.8309	27.5	208	.2200				
5.368022	16.5006	19.7	149	.1127				
5.282104	16.7709	42.5	321	.2000				
4.977751	17.8044	23.6	178	.1800				
4.672452	18.9782	32.7	247	.2800				
4.411916	20.1102	34.8	263	.0800				
4.257688	20.8467	34.2	258	.2600				
3.761157	23.6360	13.3	100	.0400				
3.699455	24.0361	22.3	168	.1400				
3.558431	25.0037	14.8	112	.1000				
3.439070	25.8864	21.0	159	.1400				
3.272550	27.2283	22.1	167	.2800				
3.218939	27.6907	17.0	129	.1400				
3.140884	28.3931	17.2	130	.0800				
3.013536	29.6198	13.9	105	.1000				
2.873482	31.0991	15.1	114	.0200				
2.782802	32.1395	16.6	125	.0200				
2.754830	32.4748	20.2	152	.0600				
2.713309	32.9858	15.4	116	.1127				

Table 2: Crystalline calcium salt of 5-methyl-(6S)-tetrahydrofolic acid (Type II)

Diffractometer : Transmission
 Monochromator : Curved Ge(111)
 Wavelength : 1.540598 Cu
 Detector : Linear PSD
 Scan Mode : Debye-Scherrer / Moving PSD / Fixed omega
 2Theta scan

! Peak search parameters : Expected halfwidth : .150
 ! Significance level : 2.5
 ! Peak height level : 10

Peaklist [Range 1 : 2Theta = 5.000 34.980 .020 Imax = 526]

!	D	2Theta	I(rel)	I(abs)	FWHM	h	k	l
	12.720530	6.9434	100.0	517	.2600			
	8.508053	10.3891	29.4	152	.2400			
	6.631466	13.3409	19.6	101	.1200			
	5.863504	15.0461	71.2	368	.2200			
	5.580025	15.8696	27.8	144	.0800			
	5.010988	17.6854	42.5	220	.1400			
	4.730443	18.7434	53.6	277	.1400			
	4.215807	21.0561	35.5	184	.0400			
	3.943879	22.5263	38.8	201	.3600			
	3.581969	24.8368	24.8	128	.0200			
	3.493985	25.4726	29.6	153	.0400			
	3.309171	26.9212	22.7	117	.0200			

Table 3: Crystalline calcium salt of 5-methyl-(6S)-tetrahydrofolic acid (Type III)

Diffractometer : Transmission
 Monochromator : Curved Ge(111)
 Wavelength : 1.540598 Cu
 Detector : Linear PSD
 Scan Mode : Debye-Scherrer / Moving PSD / Fixed omega
 2Theta scan

! Peak search parameters : Expected halfwidth : .150
 ! Significance level : 2.5
 ! Peak height level : 10

Peaklist [Range 1 : 2Theta = 5.000 34.980 .020 Imax = 817]

!	D	2Theta	I(rel)	I(abs)	FWHM	h	k	l
	12.933490	6.8289	100.0	786	.1200			
	11.036740	8.0043	18.9	149	.0400			
	9.945525	8.8842	18.4	145	.1000			
	8.877709	9.9554	12.4	98	.0796			
	8.640580	10.2293	49.6	390	.1000			
	7.573330	11.2292	6.4	50	.1000			
	7.144004	12.3799	7.6	59	.0800			
	6.948557	12.7295	20.3	159	.1000			
	6.659956	13.2835	10.1	80	.0400			
	6.466239	13.6834	7.6	60	.0200			
	6.305060	14.0349	37.6	296	.1000			
	6.154434	14.3802	16.4	129	.0400			
	6.057193	14.6123	15.3	121	.0600			
	5.920458	14.9517	17.6	139	.1000			
	5.738533	15.4285	48.9	385	.1000			
	5.530167	16.0136	30.3	238	.1000			
	5.322477	16.6428	18.1	143	.0600			
	5.245302	16.8894	47.4	372	.0800			
	5.154604	17.1888	20.9	164	.0796			
	5.038273	17.5888	30.8	242	.1000			
	4.980502	17.7945	10.7	84	.0796			
	4.759336	18.6286	31.6	248	.1200			
	4.702846	18.6544	24.3	191	.0796			
	4.575841	19.3827	15.6	122	.0800			
	4.478961	19.8061	25.9	204	.1000			
	4.377158	20.2716	48.1	378	.1000			
	4.309006	20.5957	11.9	93	.0796			
	4.242777	20.9207	31.3	246	.0800			
	4.051441	21.9207	10.3	81	.0200			
	3.940356	22.5467	67.8	533	.1200			
	3.782452	23.5010	12.4	98	.0400			
	3.609291	24.6458	9.5	75	.0200			
	3.523157	25.2582	27.0	212	.2000			
	3.460874	25.7205	43.4	341	.0800			
	3.408545	26.1223	12.4	98	.0796			
	3.341048	26.6596	16.1	127	.2000			
	3.273575	27.2196	28.4	223	.1400			
	3.188038	27.9645	12.6	99	.0200			
	3.160110	28.2168	12.5	98	.0400			
	3.103472	28.7427	15.0	118	.0800			
	3.052658	29.2317	13.9	109	.0600			
	3.017419	29.5808	27.7	218	.1400			
	2.970195	30.0621	10.6	83	.1200			
	2.921067	30.5800	13.9	109	.0200			
	2.899222	30.8161	9.6	76	.0796			
	2.870572	31.1314	9.6	75	.0400			
	2.830661	31.5817	11.0	86	.0200			
	2.758126	32.4349	11.3	89	.0400			
	2.733265	32.7382	13.2	104	.0600			
	2.695836	33.2058	13.7	108	.0800			
	2.660160	33.6643	11.7	92	.1000			
	2.609572	34.3369	9.2	72	.0200			

Table 4: Crystalline calcium salt of 5-methyl-(6S)-tetrahydrofolic acid (Type IV)

Diffractometer : Transmission
 Monochromator : Curved Ge(111)
 Wavelength : 1.540598 Cu
 Detector : Linear PSD
 Scan Mode : Debye-Scherrer / Moving PSD / Fixed omega
 2Theta scan

! Peak search parameters : Expected halfwidth : .150
 ! Significance level : 2.5
 ! Peak height level : 10

Peaklist [Range 1 : 2Theta = 5.000 34.980 .020 Imax = 473]

!	D	2Theta	I(rel)	I(abs)	FWHM	h	k	l
	13.398610	6.5916	97.7	446	.1600			
	12.930100	6.8307	100.0	457	.0915			
	11.033220	8.0069	19.2	88	.0800			
	9.952926	8.8776	16.7	76	.1200			
	8.912272	9.9167	25.5	116	.1600			
	8.626970	10.2455	48.9	223	.0800			
	6.931997	12.7600	37.4	171	.1000			
	6.651761	13.3000	39.7	181	.1200			
	6.499623	13.6127	32.8	150	.0800			
	6.309299	14.0254	47.0	215	.1600			
	6.161306	14.3641	25.1	115	.1200			
	5.917463	14.9593	27.0	124	.1000			
	5.736254	15.4347	49.8	227	.0800			
	5.544314	15.9724	36.7	168	.1600			
	5.255854	16.8553	62.1	284	.2400			
	5.172075	17.1303	29.5	135	.0915			
	5.035719	17.5978	37.0	169	.1200			
	4.978813	17.8006	31.3	143	.0400			
	4.758441	18.6321	40.7	186	.1000			
	4.688853	18.9112	46.0	210	.0915			
	4.577465	19.3757	29.5	135	.0915			
	4.479376	19.8043	35.5	162	.1000			
	4.383704	20.2410	63.6	290	.1200			
	4.246196	20.9037	59.5	272	.1400			
	4.088125	21.7216	19.7	90	.0200			
	3.941748	22.5386	62.9	288	.1400			
	3.778991	23.5229	27.9	128	.0400			
	3.696576	24.0551	30.5	139	.1000			
	3.523769	25.2537	35.6	163	.2400			
	3.459683	25.7295	44.7	204	.0800			
	3.338511	26.6803	28.7	131	.0200			
	3.273450	27.2206	45.5	208	.1000			
	3.135320	28.4446	23.6	108	.0600			
	3.108154	28.6985	25.9	118	.0200			
	3.018687	29.5681	34.4	157	.1400			
	2.923031	30.5589	21.9	100	.0200			
	2.844431	31.4249	18.4	84	.0200			
	2.749393	32.5408	28.5	130	.1200			
	2.713739	32.9804	25.6	117	.0200			
	2.663207	33.6246	19.6	90	.0600			
	2.613490	34.2838	17.4	80	.0200			